

Uranium Extraction Mechanism from Analytical Grade Phosphoric Acid Using D₂EHPA and Synergistic D₂EHPA-TOPO Mixture

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Abstract- This paper reports the extraction of uranium from AR [analytical grade] ortho phosphoric acid by D₂EHPA [di-2 ethyl hexyl phosphoric acid] only and D₂EHPA-TOPO [tri-n-octyl phosphine oxide] mixture. The influence of various factors affecting the extraction mechanism such as phosphoric acid, D₂EHPA and TOPO concentrations have been investigated. The effect of temperature on extraction process was studied and the activation energy of the reaction was calculated and found to be equal 23.1 KJ/mol. Finally, the uranium extraction mechanism by using D₂EHPA and D₂EHPA/TOPO synergism was suggested.

Keywords- *Phosphoric Acid; Solvent Extraction; Uranium; Mechanism*

I. INTRODUCTION

This document directs attention to phosphate deposits as a potential source for uranium for several reasons, e.g. the great demand for this metal, the shortage of uranium supply forecast, as well as pollution associated with the use of uranium contained phosphate fertilizers [1]. Industrial phosphoric acid is mostly produced by reaction of fluoroapatite deposit with concentrated sulfuric acid in the so called wet process. The main disadvantage of dihydrate (DH) is the production of a relatively diluted phosphoric acid which must be subsequently concentrated by evaporation before feeding in the fertilizer industries [2].

The hemihydrate process (HH) operates at 100°C and yields 38-42 % P₂O₅ phosphoric acid. This process offers a potential advantage over the dehydrate process where it can be directly used in many industries such as diammonium phosphate manufacture, sodium tripoly phosphate and dicalcium phosphate [3]. In the dehydrated process, about 80-90 % of uranium are dissolved in the produced phosphoric acid and the rest precipitates in the phosphogypsum. The extraction of uranium by organic solvent has been applied on commercial scale with many organophosphoric acids such as D₂EHPA using TOPO as synergistic agent [4-8], D₂EHPA-TAPO as synergistic agent [9], PN-1200 [supplied by All-Russian Research Institute of Chemical Technology] [10], DNPPA [dinonyl phenyl phosphoric acid]-TBP [tri butyl phosphate] [11], and in our previous study we show the kinetic studies for uranium extraction using D₂EHPA-DBBP [di-butyl butyl phosphonate] as synergistic agent [12], and PC88A [(2-ethyl hexyl) phosphonic acid, mono (2-ethyl hexyl) ester] with DBBP as also synergistic agent [13]. Although the extraction properties of the D₂EHPA-TOPO couple, which is the most conventional and practically accepted extractant composition, have been studied by a number of investigators [14-18], the synergism mechanism has not been well understood. Synergistic extraction of species with the mixture of acidic and neutral solvents was

explained by the mechanism of addition, substitution, and solvation [19-23], but the information given about the parameters affecting the synergism mechanism of D₂EHPA-TOPO couple is limited. The aim of this work is to study the relevant factors affecting the extraction mechanism of uranium from uranium synthetic AR phosphoric acid, 42% P₂O₅, by D₂EHPA and D₂EHPA together with TOPO as a synergistic agent in kerosene as diluent.

II. EXPERIMENTAL

A. Material and Reagents

All reagents used were of analytical reagent grade except D₂EHPA and TOPO manufactured by Aldrich AG which were of a commercial grade and used without purification. Kerosene was obtained from Misr petrol. Ltd., Egypt. Crystalline uranyl nitrate hexahydrate was an AR product of Fluka, Switzerland. It is used for the preparation of all uranium solutions. Uranium concentration was determined by ICP-MS and UV-VIS spectrophotometer, applying the procedure reported by Gorecka [24]. AR ortho phosphoric acid solution contains 42% P₂O₅ prepared from AR ortho phosphoric acid 85% P₂O₅ supplied from Merck, Germany. Concentration of AR ortho phosphoric acid used in this study is 42% P₂O₅ and uranium concentration is 100 ppm.

B. Experimental Procedure

The utilized organic extracts were composed of D₂EHPA and D₂EHPA-TOPO in kerosene. These phases were brought into contact with 42% P₂O₅ AR ortho phosphoric acid for 5 minutes. The extraction method was based on high dispersion in a separatory funnel by mechanical shaking. The two phases were brought into contact at ambient temperature using organic to aqueous volume ratio (R_{org/aq}) equal 1. The results were evaluated in terms of the extraction distribution ratio (D), given by the relation

$$\frac{\text{concentration of uranium in organic phase}}{\text{concentration of uranium in aqueous phase}} \times \frac{\text{volume of aqueous phase}}{\text{volume of organic phase}}$$

III. RESULTS AND DISCUSSION

A. Effect of Phosphoric Acid Concentration

The extraction of uranium from different concentration of AR ortho phosphoric acid in the range of (9-5.8 M) by D₂EHPA only and D₂EHPA-TOPO mixture was investigated and the results are given in Figs. 1, 2 as a relation between log D vs. log phosphoric acid molarity. From the figures it's clear that the extraction of uranium decreases with the increase in the phosphoric acid concentration, since it is well known that

increasing phosphoric acid concentration, the H⁺ concentration is increasing and it is also known that D₂EHPA extraction mechanism depends on releasing H⁺; therefore [12] by increase H⁺ concentration in the medium, H⁺ releasing process of the solvent is decreasing.

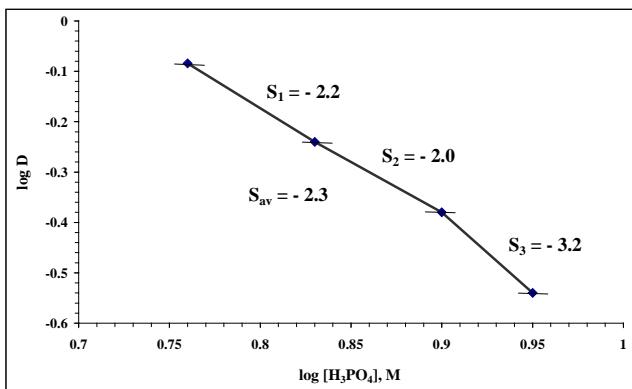


Fig. 1 Effect of ortho phosphoric acid concentration at constant D₂EHPA concentration (1.0 M)

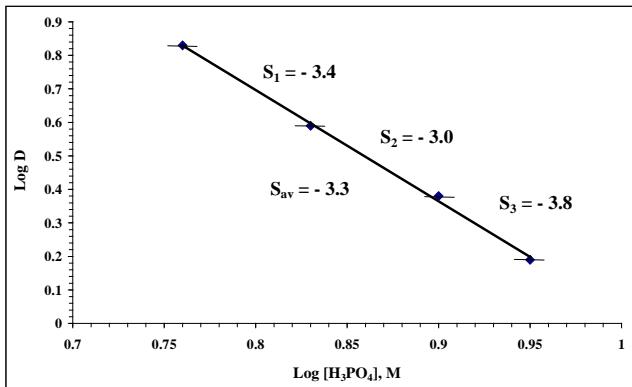


Fig. 2 Effect of ortho phosphoric acid concentration at constant D₂EHPA (1.0 M) & TOPO (0.1 M) concentrations

A mathematical average negative slope of 2.3, for D₂EHPA, and 3.3, for D₂EHPA-TOPO, was obtained from the whole acid concentration range. Therefore it can be indicated that 2 and 3 moles of phosphoric acid are liberated for extraction 1 mole of uranium from ortho phosphoric acid by D₂EHPA and D₂EHPA-TOPO mixture respectively, which implies that TOPO increases the cation exchange reaction of D₂EHPA. These results differ from the results obtained by other D₂EHPA-TOPO [8, 11] where in these systems the D₂EHPA concentration is lower than 1 M and as the extractant concentration increases, cation exchange mechanism starts to become effective [25].

B. Effect of D₂EHPA Concentration

The effect of D₂EHPA concentration on the extraction of uranium from 8.0 M ortho phosphoric acid is illustrated in Fig. 3 as a relation between log D vs. log D₂EHPA concentration. The experimental results show that with the increase in D₂EHPA concentration the extraction distribution ratio increases. Slope analysis of these results obtains an average slope of 1.75. Therefore it can be expected that 2 moles of diametric D₂EHPA are participating in the uranium extracted from ortho phosphoric acid. This result differs than other D₂EHPA-TOPO systems [25], where these systems apply for di-hydrate phosphoric acid which concentration is about 28-32 % P₂O₅.

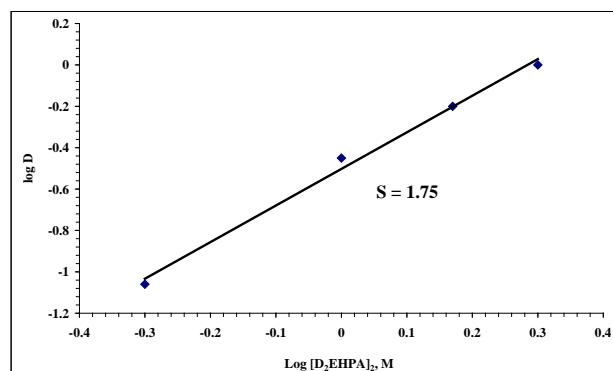


Fig. 3 Effect of D₂EHPA concentration in uranium extraction from 8.0 M ortho phosphoric acid

C. Effect of D₂EHPA Concentration at Constant TOPO

The behavior of D₂EHPA concentration on the extraction of uranium from 8.0M ortho phosphoric acid in the presences of 0.1M TOPO is demonstrated in Fig. 4 as a relation between log D vs. log D₂EHPA concentration. The experimental results indicate that the extraction increases with the increase in D₂EHPA concentration. This behavior differs from D₂EHPA alone which increases up to 1.5 M. Slope analysis of these results indicates an average slope of 1.76 for ortho acid. Further one can suggest that on the average two moles of D₂EHPA dimer are participating in uranium extraction from ortho phosphoric acid. It's clear that the number of D₂EHPA moles which participate in uranium extraction from ortho phosphoric acid is the same in absence of TOPO and in presence of TOPO.

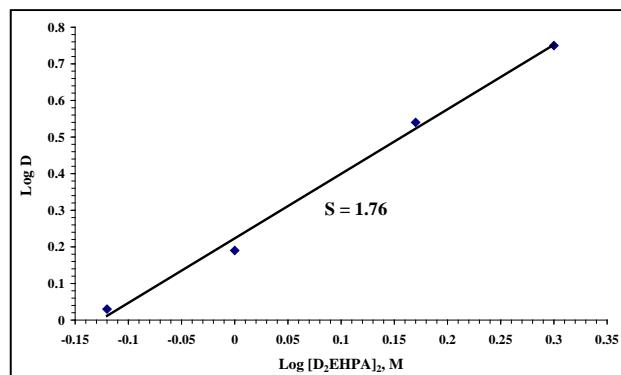


Fig. 4 Effect of D₂EHPA concentration in uranium extraction from 8.0 M ortho phosphoric acid at constant TOPO concentration (0.1 M)

D. Effect of TOPO Concentration at Constant D₂EHPA

The synergistic effect of TOPO concentration on the extraction of uranium from 8.0 M ortho phosphoric acid is shown in Fig. 5, as a relation between log D vs. log TOPO concentration. The experimental results show that the extraction distribution ratio increases with increase in TOPO concentration. Slope analysis of these results indicated an average slope of 0.82 which means that 1 mole of TOPO participate with uranium in the extracted species. This is similar to the extraction of uranium in other D₂EHPA & TOPO systems [8, 6, 25-26]. It was observed from the results that addition of TOPO to the organic phase increased the efficiency of the overall operation. It is generally accepted that the neutral organic compounds as TOPO bond to the extracted complexes through the phosphoryl oxygen atom [26].

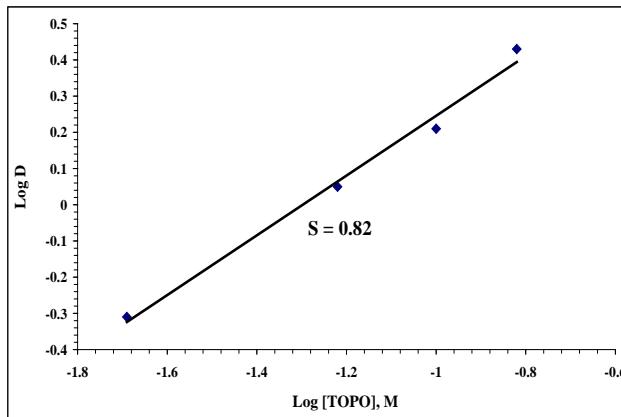


Fig. 5 Effect of TOPO concentration in uranium extraction from 8.0 M ortho phosphoric acid at constant D₂EHPA concentration (1 M)

E. Effect of Temperature

The extraction of uranium from ortho phosphoric acid, 9.16 M, at different temperature was investigated. The extraction experiments were carried out by contacting ortho phosphoric acid with a D₂EHPA-TOPO mixture, 1.0M and 0.1M respectively, in kerosene for 5.0 minute while the aqueous / organic phase ratio was fixed at 1.0 but the temperature was varied between 20 and 40 °C.

The results are presented in Fig. 6 as a relation between temperature and uranium extraction distribution ratio. From the obtained data, it can be noticed that the uranium extraction distribution ratio decreases in all cases with the increase of temperature, which indicates that the extraction of uranium from 8.0 M ortho phosphoric acid with a D₂EHPA-TOPO mixture, 1.0 M and 0.1 M respectively, in kerosene is an exothermic reaction. This result is similar to uranium extraction from phosphoric acid by other D₂EHPA & TOPO systems [6, 8].

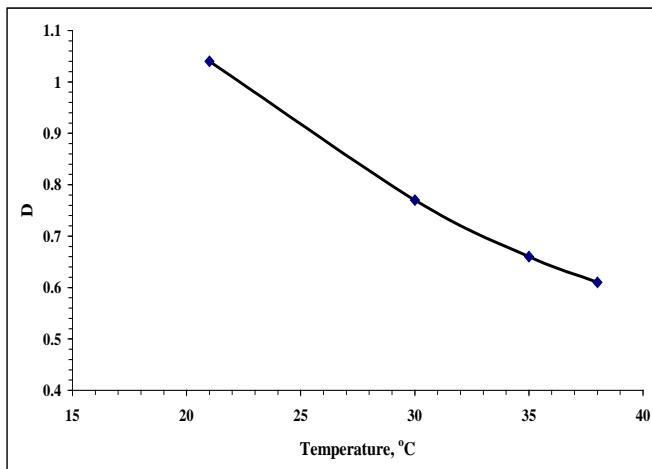


Fig. 6 Effect of temperature in uranium extraction from 8.0 M ortho phosphoric acid at constant D₂EHPA (1.0 M) & TOPO (0.1 M) concentrations

According to Van't Hoff equation, the enthalpy of uranium extraction from 8.0 M ortho phosphoric acid with a D₂EHPA-TOPO mixture, 1.0 M and 0.1 M respectively, in kerosene could be calculated by plotting relation between $\ln D$ against $1/T$ as shown in Fig. 7. From the figure, the enthalpy of uranium extraction from 8.0 M ortho phosphoric acid was calculated and found to be equal to 23.1 KJ/mol.

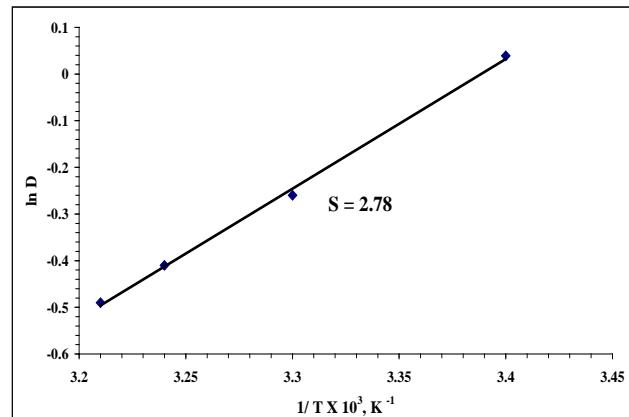
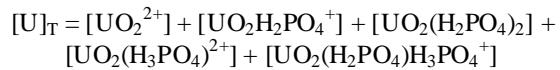


Fig. 7 Van't Hoff equation plot for uranium extraction from 8.0 M ortho phosphoric acid at constant D₂EHPA (1.0 M) & TOPO (0.1 M) concentrations.

F. Proposed Chemical Equilibrium

Studying the uranium extraction mechanism from uranium synthetic AR phosphoric acid and proposing a chemical equilibrium for the uranium extraction by each of D₂EHPA only and D₂EHPA-TOPO systems were carried out. The main equilibrium involved is based on the different uranium species in H₃PO₄ (42.0% P₂O₅). In a critical review on the chemical thermodynamic of uranium [27], the chemical equilibrium of UO₂²⁺ in phosphoric acid was reviewed, assessed and evaluated. After a comprehensive study of the different published work on UO₂²⁺ species present in phosphoric acid of high concentration, they come to the conclusion that the main equilibrium evolved is the following:

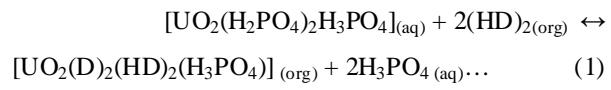


where $[U]_T$ is the total uranium concentration in phosphoric acid.

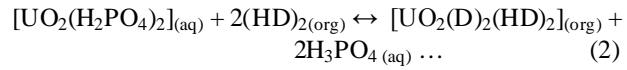
Also the following uranium species are reported [25]: UO₂(H₂PO₄)⁺, UO₂(H₂PO₄)₂, UO₂(H₂PO₄)₂, H₃PO₄ and $[UO_2(H_2PO_4)a]^{2-a}$ in pure phosphoric acid. In commercial phosphoric acid, the same species were proposed together with UO₂SO₄ and / or $[UO_2(SO_4)b]^{2-b}$ where (a and b) are integral numbers [1].

G. Proposed chemical equilibrium of D₂EHPA System

Based on the average negative slope of 2 obtained for the effect of phosphoric acid concentration on the extraction of uranium Fig. 1, the average positive slope of 2 for the extraction dependence on the D₂EHPA concentration Figure (3) and the expectable extracted uranium species $[UO_2(H_2PO_4)_2]$ and $[UO_2(H_2PO_4)H_3PO_4]$, the following equilibrium can be proposed.



and



Where (HD)₂ is D₂EHPA.

From Equation (1), the apparent extraction equilibrium constant k was calculated by the relation.

$$k = \frac{[\text{UO}_2(\text{D})_2(\text{HD})_2(\text{H}_3\text{PO}_4)]_{(\text{org})} \cdot [\text{H}_3\text{PO}_4]_{(\text{aq})}^2}{[\text{UO}_2(\text{H}_2\text{PO}_4)_2\text{H}_3\text{PO}_4]_{(\text{aq})} \cdot [(\text{HD})_2]_{(\text{org})}^2}$$

Since $D = [\text{UO}_2(\text{D})_2(\text{HD})_2(\text{H}_3\text{PO}_4)]_{(\text{org})} / [\text{UO}_2(\text{H}_2\text{PO}_4)_2\text{H}_3\text{PO}_4]_{(\text{aq})}$, then $k = D [\text{H}_3\text{PO}_4]^2 / [(\text{HD})_2]^2$. Taking the logarithm for both sides,

$$\log k = \log D + 2\log[\text{H}_3\text{PO}_4] - 2\log[(\text{HD})_2] \dots \quad (3)$$

Mathematical treating for Equation (2) as in Equation (1) yields the following relation for the apparent extraction equilibrium constant \bar{K}_1 :

$$\log \bar{K}_1 = \log D + 2\log[\text{H}_3\text{PO}_4] - 2\log[(\text{HD})_2] \dots \quad (4)$$

From Relation 3 and 4 it is clear that both of the apparent extraction equilibrium constants k and \bar{K}_1 are the same and their logarithms were calculated from all the experimental results and found to equal 1.39 ± 0.07 . This value was used to calculate the corresponding distribution ratio. It is found that, there is a slight deviation in the value of $\log D$ within the experimental error as shown in Table I, which can justify our proposed extraction equilibrium.

TABLE I EFFECT AVERAGE K AND CALCULATED CORRESPONDING DISTRIBUTION RATIO BASED ON PROPOSED EXTRACTION EQUILIBRIUM FOR URANIUM EXTRACTION FROM 8.24 M PURE H₃PO₄ USING 1.0 M D₂EHPA IN KEROSENE

$\log [\text{H}_3\text{PO}_4], \text{M}$	$\log D (\text{exp.})^*$	$\log k$	$\log D (\text{cal.})^{**}$
0.95	-0.54	1.36	-0.51
0.90	-0.38	1.42	-0.41
0.83	-0.24	1.42	-0.27
0.76	-0.08	1.44	-0.13
$\log [\text{D}_2\text{EHPA}]_2, \text{M}$	$\log D (\text{exp.})^*$	$\log k$	$\log D (\text{cal.})^{**}$
-0.30	-1.06	1.44	-1.11
0.0	-0.45	1.45	-0.51
0.17	-0.2	1.36	-0.17
0.30	0	1.30	0.09
$\log k_{av} = 1.39 \pm 0.07$			

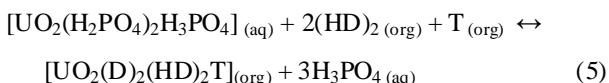
*Experimental distribution ratio.

** Calculated based on the value of k obtained.

From the previous calculations based on the extraction of two main species of uranium in AR ortho phosphoric acid, it is rather difficult to recommend certain species responsible for uranium extraction where the values of k and \bar{K}_1 obtained are the same. Therefore, we can conclude that both species contribute to the extraction of uranium from AR ortho phosphoric acid by D₂EHPA only system.

H. Proposed Chemical Equilibrium for D₂EHPA and TOPO Mixture System

Based on the average negative slope of 3 obtained for the effect of phosphoric acid concentration on the extraction of uranium in Fig. 2, the average positive slop of 2 for the extraction dependence on the D₂EHPA concentration in Fig. 4, the average positive slope of 1 from the extraction dependence on the TOPO concentration in Fig. 5 and the expectable extracted uranium species [UO₂(H₂PO₄)₂ H₃PO₄], the following equilibrium can be proposed:



where (HD)₂ is D₂EHPA. From Equation (5), the apparent extraction equilibrium constant was calculated by the relation;

$$k = \frac{[\text{UO}_2(\text{D})_2(\text{HD})_2\text{T}]_{(\text{org})} \cdot [\text{H}_3\text{PO}_4]_{(\text{aq})}^3}{[\text{UO}_2(\text{H}_2\text{PO}_4)_2\text{H}_3\text{PO}_4]_{(\text{aq})} \cdot [(\text{HD})_2]_{(\text{org})}^2 \cdot [\text{T}]_{(\text{org})}}$$

Since $D = [\text{UO}_2(\text{D})_2(\text{HD})_2\text{T}]_{(\text{org})} / [\text{UO}_2(\text{H}_2\text{PO}_4)_2\text{H}_3\text{PO}_4]_{(\text{aq})}$ then $K = D[\text{H}_3\text{PO}_4]^3 / [(\text{HD})_2]^2 \cdot [\text{T}]$, and by taking the logarithm for both sides, we get equation:

$$\log K = \log D + 3\log[\text{H}_3\text{PO}_4] - \log[\text{T}] - 2\log[(\text{HD})_2] \quad (6)$$

From Equation (6) the logarithm of the apparent extraction equilibrium constant was calculated from all the experimental results and found to equal 4.08 ± 0.11 . This value was used to calculate the corresponding distribution ratio. It is found that, there is a slight deviation with the experimental error at high D₂EHPA concentration, as shown in Table II, which can justify our proposed extraction equilibrium. The values of apparent equilibrium constant logarithm of K , in D₂EHPA-TOPO mixture system, is higher than the apparent extraction equilibrium constant logarithm of K , in D₂EHPA only system, which means that the presence of the neutral reagent TOPO increase the cation exchange reaction of D₂EHPA. Therefore, the presence of TOPO brings about important enhancement for uranium extraction from AR ortho phosphoric acid.

TABLE II EFFECT AVERAGE K AND CALCULATED CORRESPONDING DISTRIBUTION RATIO BASED ON PROPOSED EXTRACTION EQUILIBRIUM FOR URANIUM EXTRACTION FROM 8.24 M PURE H₃PO₄ USING 1.0 M D₂EHPA & 0.1 M TOPO MIXTURE IN KEROSENE

$\log [\text{H}_3\text{PO}_4], \text{M}$	$\log D (\text{exp.})^*$	$\log k$	$\log D (\text{cal.})^{**}$
0.95	0.19	4.04	0.23
0.90	0.38	4.08	0.38
0.83	0.59	4.08	0.59
0.76	0.83	4.11	0.8
$\log [\text{D}_2\text{EHPA}], \text{M}$	$\log D (\text{exp.})^*$	$\log k$	$\log D (\text{cal.})^{**}$
-0.12	0.03	4.06	-0.01
0.0	0.19	4.04	0.23
0.17	0.54	4.05	0.57
0.30	0.75	4.00	0.83
$\log [\text{TOPO}], \text{M}$	$\log D (\text{exp.})^*$	$\log k$	$\log D (\text{cal.})^{**}$
-1.69	-0.31	4.23	-0.46
-1.22	0.05	4.12	0.01
-1	0.21	4.06	0.23
-0.82	0.43	4.10	0.41
$\log K_{av} = 4.08 \pm 0.11$			

*Experimental distribution ratio.

** Calculated based on the value of K obtained.

IV. CONCLUSIONS

The aforementioned results indicate that uranium extraction from AR ortho phosphoric acid, 42 % P₂O₅, increases by increasing the concentration of both D₂EHPA and TOPO and decreases by increasing of acid concentration and temperature. The extraction process is an exothermic reaction with activation energy of 23.1 KJ/mol. For D₂EHPA system, the expectable extracted uranium species in 8.24 M phosphoric acid are [UO₂(H₂PO₄)₂ H₃PO₄] and [UO₂(H₂PO₄)₂], while the apparent equilibrium constant logarithm for both is the same and equal to 1.39 ± 0.07 .

In D₂EHPA-TOPO system the expectable extracted uranium species in 8.24M phosphoric acid are [UO₂(H₂PO₄)₂ H₃PO₄] and the apparent equilibrium constant logarithm is 4.08 ± 0.11 . The results reveal that the uranium extraction efficiency increases by using D₂EHPA-TOPO system rather than D₂EHPA alone, for that TOPO modifies D₂EHPA by increasing the transfer of uranium into the organic phase.

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